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Movement of Dislocations in Quartz

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Electronics Technology and Devices Laboratory

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13. ABSTRACT (Maximum 200 words) <p>Dislocations in quartz crystals have been known to cause problems in the fabrication of resonators by the formation of etch channels. The etch channels are known to weaken the physical strength of quartz blanks and to reduce the yield in photolithographic production processes. While it is possible to reduce the etch channel density in quartz by post growth electro-diffusion, this does not reduce the dislocation density. It is suspected that dislocations contribute to acceleration sensitivity, thermal hysteresis, and possibly aging. The behavior of dislocations in quartz is also of interest to the fields of geophysics, seismology, and plate tectonics because it affects the underground movement of rock. Specifically, the movement of dislocations in quartz is the mechanism through which quartz can be plastically deformed. A large body of literature on the movement of dislocations in natural and cultured quartz has been published in various geophysical journals over the past thirty years. This paper is a review of this literature and its possible implications for frequency control.</p>				
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Introduction

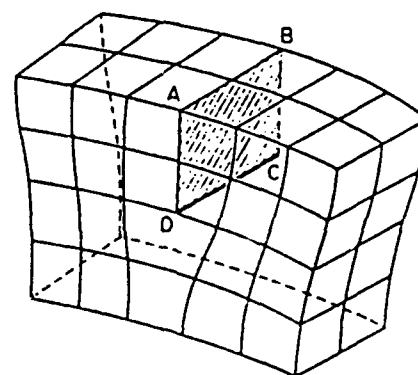
Until recently, characterizing dislocations in quartz has not been a high priority in the frequency control community. Early work indicated that dislocation densities found in cultured quartz did not adversely affect resonator performance, and, since then, most of the work has been focused on how dislocations affect the processing of resonators. Etch channels and pits, which result when quartz containing decorated dislocations is chemically etched or polished, decrease the mechanical strength of the resonators and lower the yields in manufacturing processes.^{1,2} However, as the requirements for resonator performance become more stringent, the presence of dislocations in resonators is likely to become more significant. Recent work has shown that etch channels can dramatically reduce the Q of high frequency bulk wave resonators.³ It has also been shown that the vibrational mode shape can affect the acceleration sensitivity of resonators,⁴ and X-ray topographs clearly show that mode shapes are distorted by dislocations.⁵⁻⁷ Thermal hysteresis and aging may also be affected by the presence of dislocations.^{1,8,9}

The properties of dislocations in quartz are also of interest to researchers in the fields of geology, seismology, and plate tectonics. When quartz is subjected to high stress and temperature, it flows (plastically deforms) through a mechanism involving the movement of dislocations. It has been reported that the ease with which these dislocations move is linked to the amount of water in the crystal.¹⁰ This effect has been studied extensively over the past twenty-five years, and a large body of literature has been published in the geophysical and mineralogical journals.¹¹⁻²²

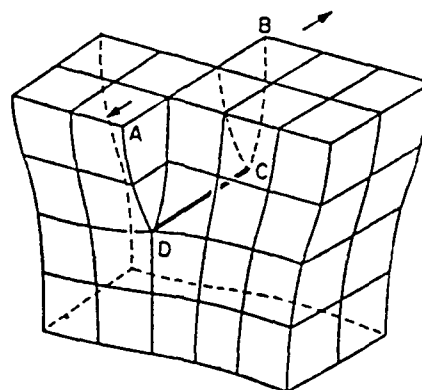
The current interest in UHF resonators, low acceleration sensitivity resonators, low hysteresis and low aging resonators, and the relatively untapped geophysical work make this an ideal time for a review paper on dislocations. In this paper we discuss historical background and current theories of dislocations in quartz. Some of the geophysical findings may be used to help explain results reported in the frequency control community.

Observing Dislocations

A dislocation is defined as a linear lattice defect. An edge dislocation is formed by inserting an extra plane



A



B

Figure 1. Edge and screw dislocations. (A) Edge dislocation, (B) screw dislocation.²³

(or planes) of atoms into an existing lattice (see Fig. 1A). A screw dislocation is generated by displacing the lattice on one side of the dislocation line relative to the other (see Fig. 1B).²³ How a given dislocation distorts a crystal lattice is given by the Burgers vector. The Burgers vector can be determined by tracing a closed circuit around the dislocation (see Fig. 2A). The path here consists of segments M to N, N to O, O to P, and P to Q, where M and Q occupy the same lattice point. Next, trace the same

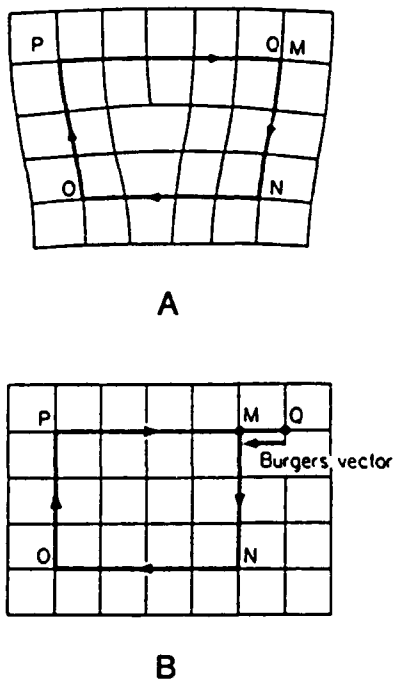


Figure 2. Determination of Burgers vector. (A) Closed circuit around a dislocation, (B) closed circuit around a perfect lattice.²³

segments on the perfect lattice, Fig. 2B. The Burgers vector is the vector Q to M required to close the circuit on M. Thus the direction and magnitude a dislocation distorts the crystal lattice is the Burgers vector. The Burgers vector of an edge dislocation is normal to the dislocation line while for a screw dislocation it is parallel.²³

There are many ways to observe dislocations in quartz, some direct and some indirect. The most common technique in the frequency control community is to use etching in order to produce etch channels.²⁴⁻²⁶ In this method a chemical etchant, usually HF, ammonium bifluoride, or a buffered oxide etchant (i.e., various mixtures of NH_4F -HF and HF) is used to etch the quartz. The etch channel density is then counted (see standard EIA-477-A²⁷). This method is indirect and insufficient in that the etch channels only form if the dislocations are decorated with impurities,^{2,26} and not all of the dislocations show up as etch channels. Sweeping moves the impurities away from the dislocation sites, and for this reason, etching will not reveal dislocations in swept quartz.

Another technique, used by Moriya and Ogawa,²⁸ is light scattering tomography. This method scans the quartz sample with a low power (in this case a 3 mW) He-Ne laser. The scattered light is then recorded on film. This apparatus is sensitive enough to observe both decorated and undecorated dislocations in quartz samples and it is non-destructive. Other methods include transmission electron microscopy (TEM)¹⁰⁻¹² and X-ray topography.^{25,29} A recent improvement on standard X-ray topography is the use of synchrotron X-rays to attain very high resolution and high speed (1 ns) pictures of dislocations and strain fields.^{5,6,30} This is especially useful for looking at the effects of dislocations on active resonators. If the quartz has been deformed sufficiently, as happens in some of the geophysical experiments, the bands of dislocations can be seen with a polarizing microscope.¹³

Impact of Dislocations on Resonators

The question of real interest for the frequency control community is how dislocations affect device fabrication and performance. First, consider the fabrication process. As mentioned earlier, when quartz containing decorated dislocations is etched, channels form. In HF, etch channels about 1 micron in diameter can propagate from the surface at more than 100 microns/hour. This is much faster than the bulk etching rate.²⁹ As stated in the previous section, sweeping can reduce the number of etch channels that form during chemical polishing and etching. However, this does not reduce the dislocation density. Hanson³¹ took X-ray topographs before and after sweeping quartz samples and no decrease was seen in the dislocations.

The next concern is the effect of dislocations on performance. A resonator property that is often measured and reported is Q. In the past it was commonly accepted that dislocations did not affect resonator Q. In 1982, Iwasaki and Kurashige²⁹ fabricated 5 MHz AT-cut resonators and found no correlation between dislocations and mechanical Q for quartz blanks with dislocation densities up to $10^3/\text{cm}^2$. In a more extensive study, Meeker and Miller³² also looked for a correlation between the dislocation density and the performance characteristics of 8 MHz fundamental AT-cut resonators cut from r-bars. They found that not only were the Q's independent of the dislocation density, but so were the resistance and inductance of the resonators. However, they did correlate the grown-in strain associated with dislocations to abnormal frequency vs. temperature (f-T) behavior. That

is, the dislocations were responsible for apparent angle shifts in resonators of up to several minutes of arc. In a recent article, Kent³ correlated Q degradation with etch channels in high frequency (45 to 140 MHz) bulk wave resonators which were chemically etched as part of the fabrication process. In these very thin resonators, a single etch channel in the active area reduced the Q by 46%. For these resonators, Kent states that low etch channel quartz (either as grown or swept) is necessary to achieve consistent Q values. Since the dislocation density is unaffected by the sweeping process, it would appear that for these resonators it is the presence of the etch channels rather than the dislocations that degrades the Q.

The apparent angle shifts in resonators found by Meeker and Miller³² suggest that dislocations can change the material constants in quartz. In support of this hypothesis, James³³ stated that he found significant differences in the elastic moduli in quartz of different grades. The difference between the grades was the level of Al impurities and the dislocation densities ($10/\text{cm}^2$ for the higher grade vs. 10^3 to $10^4/\text{cm}^2$ for the lower). Such changes in the elastic moduli would be likely to show up as deviations in the f-T characteristics (apparent angle offsets).

Recent attempts to make resonators less sensitive to acceleration have not been uniformly successful.³⁴ One reason for this seems to be that the vibrational modes are not the correct shape or in the correct place to cancel the acceleration frequency offset.⁴ Several groups have studied the mode shapes in active resonators using standard and stroboscopic x-ray topography.^{5,7} They have shown that the presence of dislocations in the active regions of the resonators distorts the mode shape, enhances coupling among modes, and interferes with mode trapping. This means that very low dislocation quartz is necessary for fabricating acceleration insensitive resonators.

Finally, consider the problems of hysteresis and aging. If dislocations change their positions during the life of the device, this could contribute to aging. Glüer et al.³⁰ saw apparent dislocation motion in stroboscopic X-ray topographs of a vibrating AT-cut resonator, but they dismissed this as an illusion caused by the vibration because they didn't believe the dislocations could be mobile at the stress levels present. In a related vein, Stephenson³⁵ used electric potentials at elevated temperatures (near the alpha-beta transition) to induce dislocations in nearly perfect quartz. He states that this

type of accelerated aging can anticipate a type of defect that might show up, in time, in resonators. If this happens gradually over months (and years) it could contribute to aging. Could dislocation movement also be a factor in hysteresis? As the temperature is increased and decreased, the dislocations may move to different potential minima, changing the crystal constants. Beaussier⁹ has put forth the proposition that slight plastic deformations, associated with the movement of dislocations, can cause an intrinsic hysteresis in bulk quartz. Certainly the quartz material appears to affect hysteresis.³⁶ But, no link to dislocation movement has been shown.

Characteristics of Dislocations in Quartz

In cultured quartz it has been observed that dislocations usually originate in the seed or at inclusions within the growth zones. It has also been observed that these dislocations propagate nearly perpendicularly to the growth front in each of the growth regions (+x, -x, s and z).²⁹ The angular distribution of dislocations in the growth zones was used by Alter and Voigt¹⁴ to show how the directions change when dislocations pass growth boundaries. They cut blanks from -x, +x, z, r, and R growth sectors and used them as seeds. The degree to which the dislocations would deviate from the growth front normal was predicted by an elastic energy minimization per length model. The dislocations on the +x seeds were nearly parallel to the seed. Meeker and Miller³² found two types of dislocations in cultured r bars. The first were uniform, slow etching, and after these dislocations had been etched into etch channels, there was no disruption of f-T curves. The second type were clustered, etched faster, and changed the f-T curves. The dislocations normally found in the z region of quartz bars are thought to be edge type and those in the +x and -x regions are screw type.^{29,37} Given that Meeker and Miller used r bars, and not z cut seeds, the two types of dislocations they observed may be edge and screw type. It is not clear, however, why these dislocations should behave so differently. Iwasaki²⁵ proposed two atomic models for the edge dislocations in quartz Z growth but the Burgers vectors of the models do not fit those determined recently by Epelboin and Patel.³⁸ X-ray topographs of Barns³⁷ were examined by Epelboin and Patel using a computer simulation method. This allowed them to determine that two different Burgers vectors are present in the Z region dislocations. These are $[1\bar{2}10]$ and $[\bar{2}110]$, with the first being the most prevalent. An example of an edge dislocation with the $[1\bar{2}10]$ vector is

shown in Fig. 3. Note that only the Si atoms have been drawn.

Over the years various methods have been tried to reduce the dislocation density in quartz.³⁹ Doherty et al.⁴⁰ attempted to grow high purity quartz. Their specific objective was low Al content and low dislocation density. By using very pure fused silica as the nutrient in gold lined vessels and growing very slowly, (0.16 to 0.35 mm/side/day) the Al content was kept well below 1 ppm (0.01 to 0.2 ppm Al) and the number of dislocations introduced during growth was reduced. To reduce the number of dislocations originating from the seed, the seeds were cut from high quality natural quartz which had been inspected by X-ray topography for dislocations. Using these methods, the dislocation density of the grown bars was kept to less than 10/cm². This material also had radiation induced offsets of less than 10⁻¹³ Hz/rad. Armington et al.⁴¹⁻⁴³ have made an extensive study of ways to reduce the number of inclusions and dislocations in cultured quartz. Some of the methods which did not improve the quartz quality were sweeping the seeds before growth, doping the seeds to change the lattice dimensions, and annealing the quartz bars. In fact, the annealing seemed to increase the strain around the inclusions. One method that did decrease the dislocation density in the

grown bars was growing on seeds cut from the +x region. Armington's data were inconclusive on the effect of silver liners on dislocations. Irvine et al.⁴⁴ at Sawyer Research also had mixed results for silver liners. The typical Sawyer quartz bar grown in a silver liner had 86 dislocations/cm², showing that simply using inert liners does not automatically abolish dislocations. Another aspect of dislocation effects was detected when Armington analyzed the material swept from quartz. They found, among other things, Al (this had also been reported by Gualtieri and Vig⁴⁵). Armington speculated that the Al was swept along dislocations since it is improbable that it would be moving through the c (z) channels.

Movement of Dislocations in Quartz

The properties of quartz, both mono-crystalline and as aggregates, have long been of interest to geologists and geophysicists. This is partly because of the abundance of this material in the earth and partly because of its unusual properties. One of the more important aspects of these is how quartz behaves at high stress levels. The long term strengths and flow properties of rock are of major importance to geophysicists because they are critical to the understanding of, e.g., earthquake seismology.¹⁵ The goal of geophysical research on the high temperature deformation of rocks is to determine the stress and strain history of the Earth's crust and mantle. This will allow accurate modeling of rock movement.¹⁶ When quartz is permanently deformed by stress, it is done through the movement of dislocations. Geophysicists are interested in using dislocation density to infer the stresses experienced by the samples while underground.

Natural quartz is unusual in the sense that the combination of high strength combined with low elastic constants means that very large elastic deformation of single crystals is possible.¹⁰ With dry natural quartz, there is a change in the response to an applied strain at around 700 degrees C.¹⁵ At this temperature there is a change that allows plastic deformation. This yield temperature is much lower for cultured (and for natural quartz that has water added during the experiment).^{10,13} These yield temperatures are dependent on the amount of water in the crystals (see Fig.-4). For cultured quartz the yield temperature ranges from 650 degrees C for 1000 ppm H/Si to 380 degrees C for 9000 ppm H/Si (here the H content was calculated from the 3 μ m IR absorption band).¹⁰ In the plastic deformation of the quartz, this movement is accomplished by dislocation propagated

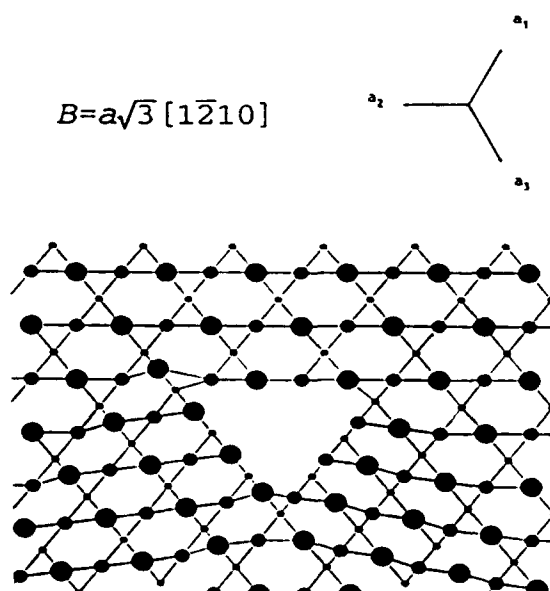


Figure 3. Edge dislocation in quartz. Note that only the Si atoms have been drawn, no O atoms are shown.

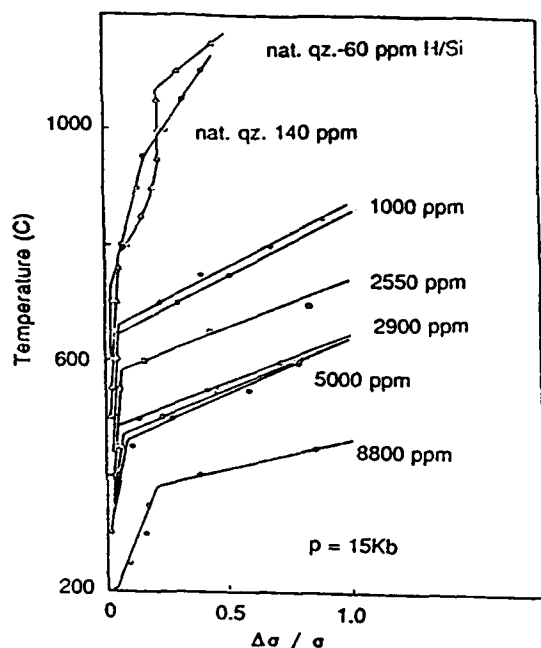


Figure 4. Fractional stress drop vs. temperature for natural and cultured quartz.

slip.^{10,13} That is, the movement of dislocations allows bulk plastic flow. The slip planes in quartz, which correspond to preferred directions for dislocation movement, have been determined.^{24,10} Tullis¹⁶ notes that there is a critical weakening pressure in the same sense that there is a yield temperature. After quartz is plastically deformed, high dislocation densities appear. Hobbs examined cultured

quartz that had been permanently deformed at 300 to 400 degrees C using TEM and found large numbers of tangled dislocation lines.¹⁷

One of the first theories which considered the role of water in dislocation movement was the Frank-Griggs model¹⁰. In the Frank-Griggs model (Fig. 5), the effect of water weakening is explained by the hydrolyzing of O-Si bonds.¹⁰ This supposedly makes the movement of the dislocations easier because the O-H bonds are easier to break than O-Si. Recently, McLaren et al.¹² looked at the problem of water weakening using TEM. They found that when wet quartz is heated, water bubbles expand and generate dislocation loops. These loops can, given time, connect several water bubbles. They claim that the OH terminations do not make the dislocations glide easier. Instead, when the quartz is heated, the bubbles expand (see Fig. 6) and generate the dislocations needed for dislocation propagated slip to occur. This explains the incubation time (the delay between the time the sample was loaded and when the first signs of strain are detected) found in some experiments. This also explains the results Armington et al.⁴³ reported after annealing cultured bars. As mentioned earlier, Armington et al. found that the strain around the inclusions was increased after annealing, and in the same paper they stated that most of the dislocations in their quartz were voids, partially filled with liquid. These may correspond to the bubbles seen by McLaren. Brice² has conducted experiments in which the alpha (the infra-red absorption coefficient at 3500 cm⁻¹, proportional to H content) is shown to be correlated to dislocation density. That is, the dislocation density can be

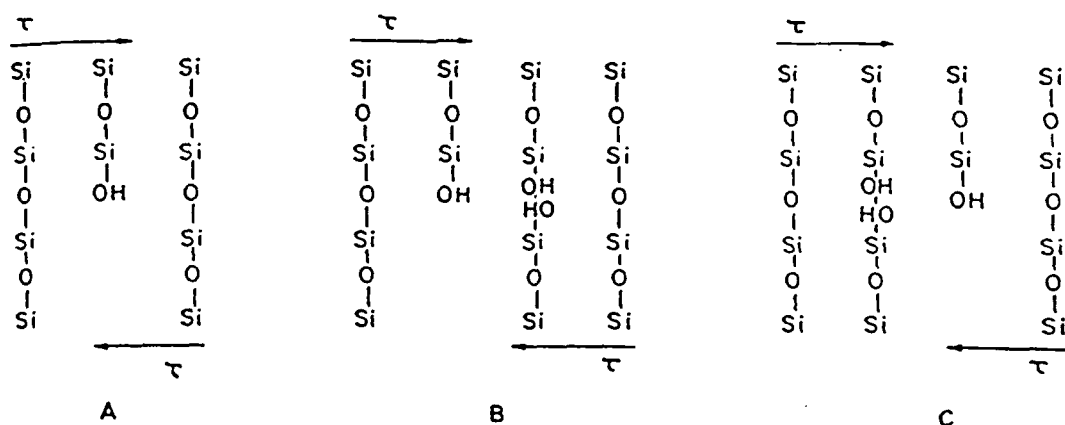


Figure 5. Frank-Griggs mechanism of dislocation movement. (A) Hydrolyzed edge dislocation with anhydrous neighbors. (B) Si-O-Si bond hydrolyzed by water migration. (C) Dislocation moves by exchange of hydrogen bond.

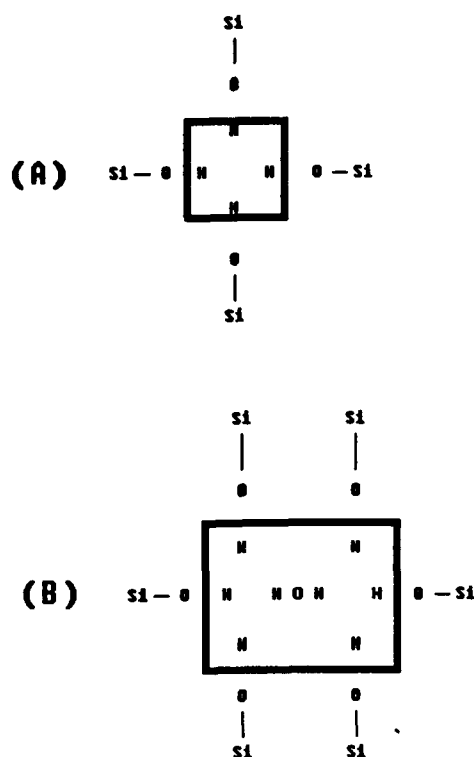


Figure 6. McLaren model of dislocation formation. (A) Bubble in quartz prior to heating. (B) Expansion of bubble through the incorporation of water.

estimated from alpha (Q_{1R}). Samples with higher alpha's (lower Q 's) have higher dislocation densities. This can be fit to the curve

$$\log_{10} N_D = 5.00 \pm 0.48 + 2.5 \log_{10} \alpha$$

where N_D is the dislocation density (cm^{-2}) and α is in cm^{-1} . Furthermore, quartz material from different manufacturers yields different lines. Brice feels that this difference in the alpha vs. dislocation density plots for quartz from different suppliers is more supportive of the Griggs-Frank model as opposed to the McLaren model.

Summary

It has been shown in the geophysical work that dislocations definitely can move in quartz. However, all of that work was done at high temperatures and pressures. It remains to be seen whether or not dislocations can be formed, or are mobile under the conditions present during the fabrication and operation of resonators. One area that was not covered in the publications reviewed here was the possibility of reducing the dislocation density through post growth pressure treatment. The possibility of moving dislocations out of the active areas in resonator blanks is very appealing and deserves to be investigated.

In parallel with these experimental studies, a theoretical basis for the behavior of dislocations in quartz needs to be developed. Heggie et al.¹⁸⁻²² have published several papers studying the dislocation lines in alpha quartz using atomic models with Keating type interatomic potentials. While this is useful for determining which dislocations are possible, it is essentially a static process. Pontikis⁴⁶ recently made the point that computer molecular dynamics (MD) techniques are very well suited for examining the movement of dislocations in crystals. With MD simulations it would be possible to better interpret the dislocation experiments suggested in the last paragraph. Also, it is possible to perform simulations of P-T conditions that would be difficult and costly to do in experiments.

Acknowledgements

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